

The durability of parylene coatings on neodymium–iron–boron magnets

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SUMMARY A parylene coating is frequently used to prevent corrosion of neodymium–iron–boron magnets when they are used intra-orally. This *in vitro* study was designed to test the durability of parylene coating in a simulated oral environment. Single and double parylene-coated magnets were subjected to grinding and crushing forces in an industrial ball mill. The results demonstrate that abrasion and wear was visible around the edges after 1 hour of testing, with a breach of the coating noted under high magnification scanning electron microscopy (SEM). The conclusion of the study is that parylene coating is unlikely to withstand intra-oral forces. The shape of the magnets, the manufacturing process involved in their production, and the thickness of the parylene coating are important factors to consider with respect to the durability of magnets used in the mouth.

Introduction

The use of magnets was reported in dentistry as early as 1960 as an aid to denture retention (Behrman, 1960). Subsequently, use of various types of maxillofacial prostheses and obturator devices incorporating magnets to facilitate their retention have been reported (Javid, 1971; Federick, 1976). The use of magnets for tooth movement was first described by Blechman and Smiley (1978) and, subsequently, magnets have been introduced into clinical orthodontic practice (Muller, 1984; Blechman, 1985; Dellinger, 1986; Kawata *et al.*, 1987; Gianelly *et al.*, 1988, 1989; Woods and Nanda, 1988; Kalra *et al.*, 1989; Sandler *et al.*, 1989; Vardimon *et al.*, 1989, 1990, 1991; Kiliaridis *et al.*, 1990; Sandler and Fearne, 1990; Sandler, 1991; Springate and Sandler, 1991; Darendeliler and Joho, 1992, 1993; Bondemark and Kurol, 1992; Darendeliler *et al.*, 1993, 1995).

Neodymium–iron–boron ($\text{Nd}_2\text{Fe}_{14}\text{B}$) magnets produce extremely high magnetic flux densities relative to their size, generating a force 16 times greater than the equivalent sized aluminium–nickel–cobalt magnet (Vardimon *et al.*, 1991). They are now accepted as the magnet of choice

in orthodontics. Although many authors have shown that these magnets have acceptable biocompatibility (Altay *et al.*, 1991; Camilleri and McDonald, 1993; Bondemark *et al.*, 1995; Bondemark and Kurol, 1997) orthodontic magnets may be in the mouth for long periods of time and have been shown to corrode (Vardimon and Muller, 1985; Blechman and Steger, 1995). Corrosion is particularly marked in the presence of bacterial plaque. Wilson *et al.* (1995, 1997) found that $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets suffered a 3.2 per cent decrease in mass over a 21-day period in the presence of a plaque biofilm. These authors, however, also showed that a parylene coating provided protection against such corrosion.

Various protective coatings have been applied to magnets in an attempt to prevent corrosion (Behrman, 1960; Tsutsui *et al.*, 1979; Blechman, 1985; Sandler *et al.*, 1989). Parylene was first suggested by Vardimon *et al.* (1991). Parylene [poly-(para)-xylene] is a conformal polymer coating used in many electrical and medical applications, and is made of 1,4-phenylene 1,2-ethenediyl. It has good physical properties, together with a very low permeability to moisture and other corrosive gases (Table 1). Parylene

Table 1 The properties of parylene.

Properties	Parylene C
Tensile strength	68.9 MN/m ²
Yield strength	55.2 MN/m ²
Elongation to break	200%
Density	1.289 g/cm ³
Coefficient of friction	0.29
Water absorption	0.06 in 24 hours

coatings are applied to rare earth magnets by vacuum deposition at an ambient temperature. The coating is created by conversion of a crystalline dimer by heat to a gaseous form. This is then converted to a monomeric gas with application of additional thermal energy. Polymerization occurs during the final deposition process. The precursor powder is first vaporized at approximately 150°C in a vacuum and the resulting dimer gas is heated to approximately 680°C to yield the monomeric diradical para-xylene. Finally, this monomer gas enters an ambient temperature deposition chamber, where it simultaneously adsorbs and polymerizes on the substrate.

The manufacturers claim that the parylene coating is of uniform thickness, pinhole free, physically stable, and chemically inert, and is therefore extremely resistant to chemical attack. It is also insoluble in most known solvents. Parylene remains stable at continuous temperatures as high as 130°C in air or 220°C in the absence of oxygen. It has good mechanical properties from -200°C to 275°C. Since polymerization of the film takes place at room temperature, there is no thermal or mechanical stress during application, and the performance parameters of the coated specimens are basically unaffected.

In clinical applications, magnets and their coatings are subjected to mechanical abrasion from tooth brushing, mastication, and sharp point scratches, as well as chemical erosion from acidic drinks or food. Previous studies have shown that occlusal forces can range from 200 to 3500 N and biting forces during mastication are in the range of 50–200 N (Craig, 1994). Any material, and even the teeth themselves, may undergo attrition or abrasion under constant

exposure to masticatory forces. A coating, therefore, may fail as a result of abrasion or scratching to such a degree that the magnet is exposed to the oral environment. Once this occurs, magnets have been shown to corrode at a rapid rate (Wilson *et al.*, 1995, 1997). It is essential that coating materials are not only biocompatible, but also strong enough to resist intra-oral forces. Although parylene appears able to protect the magnets from corrosion, no studies of the robustness of this coating material have been carried out.

This study was designed to determine:

1. the survival time of parylene coating of Nd₂Fe₁₄B magnets when subjected to simulated masticatory forces;
2. whether a double parylene coating provides better resistance than a single coating.

Materials and experimental methods

Materials

Neodymium-iron-boron (Nd₂Fe₁₄B) non-magnetized magnets of dimensions consistent with those used clinically during the management of unerupted teeth (4 mm diameter by 3 mm height) were coated with a single parylene 'C' coating (12 µm) or a double parylene coating (24 µm). Non-magnetized magnets were used to stop them sticking together within the testing apparatus, so that they could be subjected to SEM.

Apparatus

A ball mill machine powered by an electric motor (Figure 1) was used to simulate the oral environment. This has been shown to be a convenient and reproducible means of producing mechanical fatigue, and for predicting the clinical performance of dental materials (Abu Kasim *et al.*, 1996). The inner surface of the cylindrical ball mill was made up of three lifters used to lift up the grinding media during the rolling process and ensure that it fell from its highest point.

Three zones of comminution can be elicited in a ball mill: crushing, grinding, and tumbling.

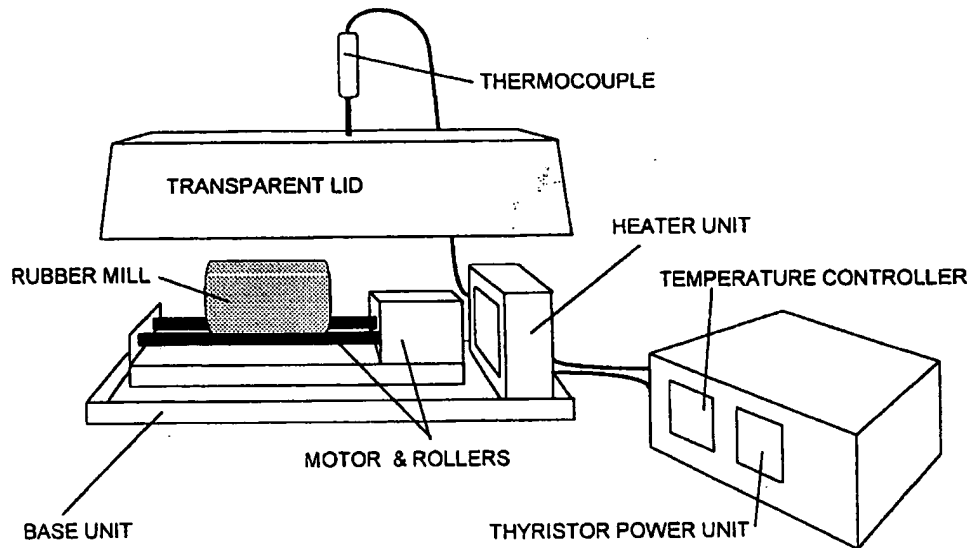


Figure 1 The test apparatus used in this investigation.

Each zone is characterized by the type of action produced by the grinding media. The crushing zone is formed by the media falling on to the material under test, thereby crushing and pulverizing it. Grinding occurs between layers of the media as they slip past one another, and includes impact or compression forces, chipping due to oblique forces, and attrition due to forces acting parallel to the surfaces. The tumbling zone is characterized by cascading media that roll over one another. Breakage in this situation may be caused by impact, but of a much lower energy level than in the crushing zone (Tarasiewicz and Radziszewski, 1990). Steatite balls with a diameter of 9.5 mm were used as the grinding media. Steatite is a ceramic material with a Vickers hardness and coefficient of friction

similar to tooth enamel, and therefore has similar wear characteristics (Wassell *et al.*, 1994a,b).

Method

Parylene-coated magnets were placed within the ball mill with a 'charge' of steatite balls. Several permutations of coating and charge were employed, and these are summarized in Table 2. In order to eliminate problems encountered with premature failure of the coating at the edges of the magnets, epoxy resin plugs were attached to the ends of the specimens. These plugs exposed only the smooth surface of the magnets to the grinding media, thereby strengthening the validity of the results.

Table 2 The testing regimes.

Test	Type and amount of media	Magnets
S1	40 steatite balls	4 single-coated
D1	40 steatite balls	4 double-coated
S2	40 steatite balls	4 single-coated with epoxy ends
D2	40 steatite balls	4 double-coated with epoxy ends
S3	450 g of steatite balls	4 single-coated with epoxy ends
D3	450 g of steatite balls	4 double-coated with epoxy ends
4	2% NaCl solution	Magnets that survived the previous tests

For each run, 250 ml of distilled water was added to the mill and the whole apparatus placed in an incubator at 37°C. The ball mill was rotated at 60 rev/min. In each test, four magnets were used. One was removed every hour, and the magnet examined visually and under SEM to determine the effects of the ball mill test on the coating.

Tests employing 40 steatite balls were designed to represent impact forces, such as would be expected with tooth-tooth impact damage, whereas 390 balls represent more grinding and shearing types of force.

For those tests where specimens did not demonstrate a breach of the coating, the test was repeated and the magnets were placed in an aerated sodium chloride solution to identify any microscopic breach.

Results

Under SEM, the surface of the untested samples appeared undulating with slightly smooth and flat edges. There were no detectable breaches in the parylene coating.

Tests

Single coating 40 steatite balls (S1). Each magnet with a single parylene coating showed signs of wear and abrasion around the edges after 1 hour, which became more obvious with time. After 2 hours, signs of tearing of the coating were already present. Finally, at 4 hours, the coating became detached from the magnet. Low magnification SEM at 1 hour showed that the surface of the parylene was dramatically smoother relative to the control. The edges became thinner and some areas displayed breakage. The edges of the parylene appeared deformed as if pushed and sheared away.

Higher magnification showed a clear break of the parylene, together with a small amount of flaking. As time increased, the coating was seen to come away from the magnet, and there was a clear space between the parylene coating and the magnet (Figure 2). At 4 hours, the whole of the parylene coating became detached from the magnet.

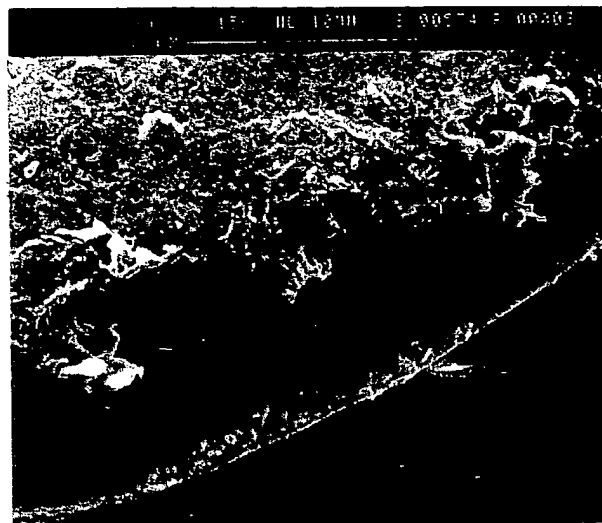


Figure 2 SEM micrograph showing the parylene coating detached from the magnet ($\times 200$ magnification).

Double coating 40 steatite balls (D1). At 1 hour, minimal changes were evident to the coating and, under SEM, the edges were flattened as if the parylene has been pressed against the edges of the magnet. High magnification showed that there was a small breach at the edge. These changes increased with time with the edges showing abrasion and tearing. At 4 hours, the whole parylene coating became detached from the magnet as the edges were completely torn. Under high magnification SEM, the inner surface of the parylene in particular showed clearly the presence of high molecular weight substances (e.g. metals) attached to it (Figure 3).

Summary of results of tests S1 and D1 (Table 3)

Single coating 40 steatite balls + epoxy ends (S2). At 1 hour, the coating appeared satisfactory, and there was no sign of abrasion or tearing of the parylene. SEM showed that, although the resin parylene junction appeared cracked, the edges were still protected. At 3 hours, the coating started to show visual signs of abrasion near the joint area. High magnification SEM confirmed that there were some small lines near the joint areas, indicating that a crack was propagating around the joint circumference. The parylene coating, therefore, was in the initial

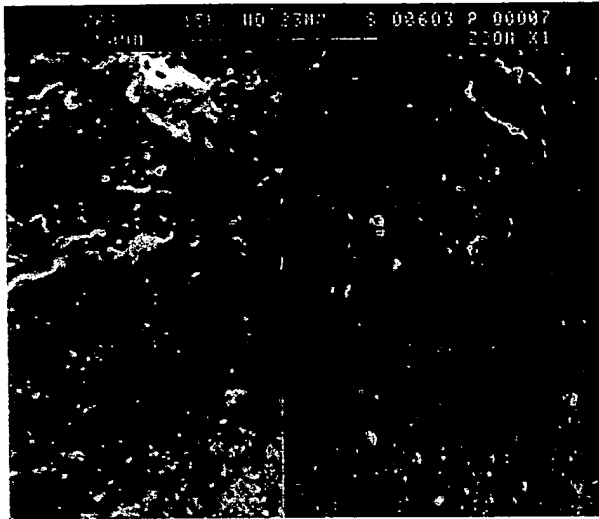


Figure 3 SEM micrograph showing magnetic material attached to the inner surface of the parylene coating ($\times 768$ magnification).

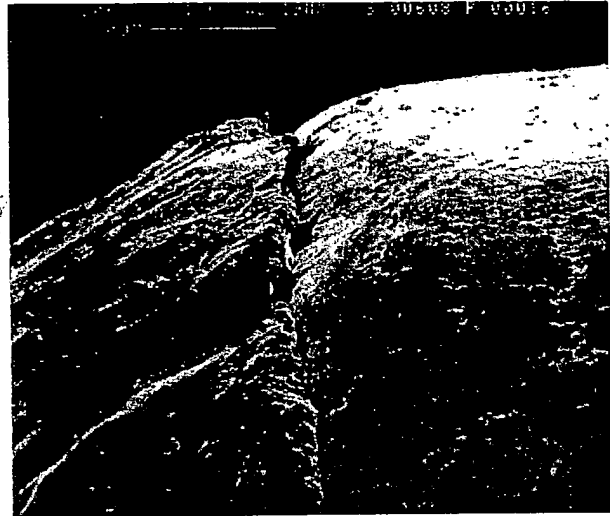


Figure 4 SEM micrograph showing the parylene coating coming loose from the magnet surface ($\times 106$ magnification).

Table 3 Summary of results of tests S1 and D1 using 40 steatite balls.

Time interval (hours)	1	2	3	4
Test S1: single coating	+	+	+	+
Test D1: double coating	+	+	+	+

+ Denotes a breach in the parylene coating.

Table 4 Summary of tests S2 and D2 using 40 steatite balls.

Time interval (hours)	1	3	5	7
S2 single coating	-	+	+	+
D2 double coating	-	-	+	+

+ Denotes a breach in the parylene coating; - denotes the parylene is intact.

stages of failure. At 5 hours, the coating appeared to have separated partially from the magnet and there was obvious tearing of the parylene around the edges. SEM showed a clear breach at the joint area with some parts of the parylene extruded beyond the joint level. At 7 hours, the separation of the coating increased and the dimensions of the resultant cylinder of parylene appeared to be larger than the original magnet. The parylene coating appeared to be loose (Figure 4). Tearing occurred around the joint area and SEM confirmed a clear breach with the parylene extruded beyond the magnet.

Double coating 40 steatite balls + epoxy ends (D2). At 1 and 3 hours, the coating appeared

satisfactory and there was no sign of obvious abrasion or a breach of the coating, either visually or under SEM in any of the specimens. At 5 hours the coating appeared to be slightly rounded and demonstrated abrasion around the edges. SEM showed there was a clear breach near the junction between the parylene and the epoxy material and this was significantly worse at 7 hours.

Summary of tests S2 and D2 (Table 4)

Single coating 450 steatite balls + epoxy ends (S3). At 1 hour, the coating appeared rounded and signs of abrasion could be seen around the edges of the magnets. High magnification showed the

presence of cracks on the surface of the coating. At 3 hours, the parylene surface appeared slightly wavy and displayed evidence of abrasion around the edges. Small amounts of water appeared to emerge from underneath the coating edges when a dry tissue was applied. At 5 hours, the coating became loose and high magnification SEM showed a clear breach present at the edge near the joint between the coating and the epoxy resin. This was more obvious at 7 hours.

Double coating 450 steatite balls + epoxy ends (D3). The parylene exhibited only minimal change at 1 hour and high magnification SEM showed no obvious breach. A brown discolouration, however, appeared under the coating. At 3 hours, abrasion around the coating became more obvious, but there was no sign of tearing. High magnification, however, showed that the edge of the coating had started to fail and small crack lines had appeared. At 5 hours, these effects were more significant and at 7 hours high magnification SEM demonstrated a clear breach in the coating.

Summary of the results of tests S3 and D3

This test was performed to determine the effectiveness of the coating types that had survived from tests S2 and D2. Fresh samples were tested in the ball mill under the same conditions for slightly shorter periods of 30, 60, 90, and 180 minutes. They were then immersed in the sodium chloride solution.

The single parylene specimen that had been tested for 30 minutes in the ball mill corroded after being in the solution for 7 days and the 60-minute specimen corroded after being immersed in the sodium chloride solution for 3 days. These results confirmed that the coatings were breached, even though this was not visible to light or scanning electron microscopy (Table 5).

The double parylene samples tested for 30, 60, and 90 minutes showed no changes after immersion in sodium chloride solution for 14 days. However, the specimen that had been tested for 180 minutes did corrode.

Table 5 Summary of the results of tests S3 and D3 using 450 g of steatite balls.

Time interval (hours)	1	3	5	7
Single coating	+	+	+	+
Double coating	+	+	+	+

+ Denotes a breach in the parylene coating.

Table 6 The effectiveness of the coatings that had survived from tests S2 and D2 after immersion in solution of sodium chloride.

Time interval (hours)	0.5	1	1.5	2
Single parylene	+	+	Not tested	Not tested
Double parylene	-	-	-	+

+ Denotes corrosion occurring; - denotes no breach in the coating.

The effectiveness of the coatings that had survived from tests S2 and D2 after immersion in sodium chloride solution (Table 6)

After a further period of 5 days in air, the uncorroded magnets from test 4 showed clear signs of discolouration under the parylene coatings. Such discolouration could not be seen in the control specimens and was therefore interpreted as evidence of corrosion.

Discussion

The ball mill is a successful method of simulating intra-oral masticatory forces and has been shown to be comparable to the clinical environment (Millett *et al.*, 1995; Abu Kasim *et al.*, 1996). The types of impact produced in the ball mill depend on the speed of rotation of the mill and the charge within it. These were carefully selected in this study to be consistent with previous reports (Tarasiewicz and Radziszewski, 1990).

The forces (and, hence, stresses) experienced by the samples are due not only to the speed of impact of the steatite medium used, but also depend strongly on the rate of deceleration of

the impacting body and, hence, on the nature of the surface coating. The high ductility and low yield stress of the parylene allow the material to deform under the impact forces experienced in the ball mill (or in the mouth). These properties, combined with the thinness of the coating, make it likely that much of the impact energy is absorbed by the magnet itself. Since the latter is made from a relatively non-ductile material, the impact forces (and stresses) on the magnet itself would be expected to be high. The magnets are sintered and the inherent porosity generated by this process can render the material brittle and easily cracked. It is not surprising, therefore, that the test specimens which exhibited complete parylene separation when viewed under SEM, revealed that it was the bond between the surface grains of the magnets that had failed, and not the bond between parylene and the magnet. This was demonstrated by the clear signs of magnet alloy particles adhering to the inside surface of the separated coatings. The thicker the parylene coating, however, the smaller the force that is experienced by the magnet. Theoretically, therefore, it should be possible to thicken the parylene layer to the extent that all the impact energy is absorbed by the coating and none is transferred to the underlying magnet. This, however, may not overcome the effects of scratching and abrasion, which were not addressed in this study.

Early tests showed signs of coating failure at the two circular edges at either end of each magnet. This is to be expected, since it is at these sites where the minimum radius of curvature and thus the maximum stress (in the system) is encountered, both when considering impact and grinding mechanisms. Once the ends of the coatings had been eroded away, subsequent failure of the specimen was rapid. It was thought that such rapid failure could preclude the observation of alternative forms of failure, such as direct penetration of the coating. In view of this, the ends of the magnets were protected using an epoxy resin cap for subsequent tests. Whilst the protection was highly effective, separation of the coating from the magnet occurred just as rapidly as for the non-protected specimens. Thus, it can be confirmed that damage to the magnet itself was the predominant failure mechanism.

The present method of testing identified that it was the magnet that was failing, rather than the adhesion of the coating on the magnet. In view of this, the ball mill can be seen as a highly appropriate method of testing as it involves the whole component, rather than just the coating. For other materials, it may be necessary to characterize the properties of the coating itself and, in those cases, alternative methods, such as scratch testing, may be required.

Although most of the ball mill tests were performed with a charge of 40 balls, a number contained approximately 390 balls. It was hoped that this substantial increase would lead to a change in the predominant mode of force delivery within the mill, from one based on impact to one based on grinding and shearing motions. The results, however, did not show a dramatic variation in the time to failure or in the mode of failure of the coated magnets. The reasons for this are unclear, but it may be that the magnets were subjected to substantial impact forces in both testing regimes or that 450-g balls were insufficient to alter the force mode. It should also be noted that steatite, being a ceramic, is relatively incompressible; therefore, an impact force will be transmitted from one steatite ball to another, with little reduction in intensity.

The final series of tests involved the submersion of the coated magnets into a corrosive aqueous solution aimed at simulating the oral environment. The main purpose of these tests was to determine whether a coated magnet, tested for only a short period of time within the ball mill and which had not shown any signs of failure, was nevertheless breached in some way. After a week of submersion no obvious signs of corrosion were visually detected in the double-coated specimens. However, under SEM a definite flattening out of the surface topography of the parylene coatings was noted. This is consistent with the arguments presented earlier regarding parylene's low yield strength and high ductility.

Despite not being able to demonstrate a breach in the coating, the double-coated specimens showed signs of corrosion after a further period of 5 days in air. The presence of corrosion

products under the coating may be explained in a number of ways; the generation of pinholes during the ball mill test, diffusion/absorption of moisture through the parylene or the generation of cavities under the layer, combined with diffusion or absorption of moisture. As the control specimens did not show any sign of this corrosion it may be that the impacting balls created fractured areas close to the coating/magnet interface. These areas provided cavities, and therefore the opportunity for both oxygen and moisture to react with the iron content of the magnet, and generate a form of crevice corrosion. The relevance of this finding is that even impacts within the oral cavity that do not generate a physical breach of the parylene coating, could eventually lead to failure of the coated magnets. This casts doubts on the effectiveness of parylene as an adequate coating for Nd₂Fe₁₄B magnets used in the oral cavity where they could be subjected to intra-oral forces.

Conclusions

1. The apparatus and method used in this investigation, proved successful in testing the durability of the parylene coating on neodymium-iron-boron magnets.
2. Parylene is a weak polymer, which readily deforms under simulated oral impact loads and parylene coatings presently used are too thin to absorb significant impact energy.
3. Increasing the thickness of the parylene layers generally improves the resistance of the coated magnets to the onset of failure.
4. The behaviour of the sintered magnetic alloy is more likely to be the underlying cause of failure of a coated magnet. Failure occurs via the progressive destruction of the sintered surface layers under impact loads.
5. Changing the design of the magnets by rounding their edges would make them less susceptible to failure of both the coating and the underlying magnet.
6. Ingress of moisture through either pinholes or by natural diffusion, combined with probable cavity generation between coating and substrate (by impact processes), causes rapid crevice corrosion, which can ultimately lead to failure of the magnet/coating system, probably even under low impact loads.

Overall, it can be concluded that the results of this study do not support the hypothesis that the present design of parylene-coated magnets makes them suitable for use in the oral cavity.

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